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# USING DIFFERENT TECHNOLOGIES TO SOLVE UNIQUE PRECISION CLEANING PROBLEMS

by

Don E. Hunt

Chief Scientist
Aerospace Guidance and Metrology Center
Newark Air Force Base, Ohio 43057-0013
United States of America

Commercial Phone: (614) 522-7712
FAX : (614) 522-7449

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## INTRODUCTION

For years those industries requiring precision cleaning in their production operations had access to chemical solvents which, because of their extraordinary properties and when used with the appropriate process and equipment, would suffice to precision clean virtually everything requiring precision cleaning. Principle among these were 1, 1,2-trichloro l,2,2-trifluoroethane (CFC-113) and methyl chloroform (MCF) . When the Montreal Protocol process identified these chemicals as ozone depleting chemicals (ODCs) and targeted them for complete phaseout, the industries requiring precision cleaning were challenged to develop and implement alternatives for CFC-113 and MCF.

Efforts began immediately and worldwide with a great deal of success. Through those efforts there are now an abundance of alternatives for virtually every precision cleaning requirement. This sounds very impressive, and it truly is. Initially it would appear that there should no longer be any difficulties replacing CFC-113 and MCF within the industries using those chemicals for precision cleaning. However, upon examination this is definitely not the case.

While it is true there are abundant alternatives, none of them are universal. For example, there is no substitute chemical that is a perfect replacement for either CFC-113 or MCF in all their applications. In addition, there are a variety of parameters associated with the alternatives that vary considerably among those alternatives. These include such things as environmental impact, cost, worker safety, flammability, storage lifetime, different levels of reactivity with different materials, and so on. To further compound the problem, these parameters vary in importance depending upon such things as the geographic area involved, type and nature of process requirements, and political concerns.

The challenge now is not to develop new alternatives; it is to find existing alternatives that may work for a given process and, then, to select the most appropriate one for adaptation. To do this, it must be recognized that an alternative is most likely not going to be a "drop-in" chemical substitute--it is more probably going to be an entirely new process! With this in mind, it is imperative that a current process using an ODC be well understood. This implies understanding the "purpose" for which the process exists, not just how the process, itself, works. Only when this has been accomplished can potential alternative processes be examined and the "best" one selected.

Once the preferred alternative is selected and ready to be tested and evaluated, it is critically important to understand that someone else perfected the alternative process for their specific needs and situation. This means that the process will undoubtedly need to be tailored, or adapted, to the new requirements. It is also important to realize that with the number of variables involved in the typical precision cleaning process, nothing ever works the first time it is tried. It will require a few iterations to be successful; it is important to approach the effort with this thought in mind.

In a diversified industrial operation with a variety of precision cleaning requirements, it is quite probable that one alternative process cannot be adapted to fit every need. In precision cleaning, every different cleaning operation often presents a "unique cleaning problem" to be resolved. More than one alternate technology may be required to solve the variety of problems that will arise.

At the United States Air Force's Aerospace Guidance and Metrology Center, it has been necessary to select, adapt, and apply several different technologies to resolve some of the unique problems that have presented themselves in the Center's quest to totally eliminate its use of ODCS for general and precision cleaning.

## BACKGROUND

The Aerospace Guidance and Metrology Center (Center) is located in the state of Ohio, USA, at the Newark Air Force Base. It is a repair center in the US Air Force Materiel Command.

One of the Center's primary missions is the repair of inertial guidance and navigation systems and components used by most missiles and aircraft in the US Air Force inventory. The inertial systems and components of several foreign countries are also repaired at the Center.

The Center's industrial operations are contained within one large building covering approximately fifteen acres (61 000 square metres). Within this building are a large number of smaller structures totalling over 200,000 square feet (18 600 square metres) of floor space. These structures have strictly controlled environments and contain a vast array of complex repair operations.

The sophisticated electromechanical devices that form the nucleus of inertial systems are extremely susceptible to minute contamination, both particulate and non-particulate residue. As a result, great care must be taken to assure a clean repair environment. Of course, during the repair process it is necessary to "precision" clean many of the parts being assembled.

The Center has historically used large quantities of CFC-113 for both general and precision cleaning. Prior to 1988 the center used over 2,750,000 pounds (1 250 000 kilograms) of CFC-113 each year. Other solvents used at the Center for cleaning include MCF and trichloroethylene. While not an ODC, trichloroethylene is a particularly toxic chemical. The Center, in its efforts to find alternatives for ozone depleting solvents, has recognized that there are other solvents in use which it should be able to eliminate with the same alternatives. Consequently, though the attention is on ozone depleting solvents, alternatives are simultaneously being sought for many other solvents classified as hazardous at the Center.

## AQUEOUS CLEANING TECHNOLOGY

When the Center began its quest for alternatives, it made a corporate decision to seek, as a first choice, a technology which would provide the most environmentally friendly solution and which would be the least likely to come under close scrutiny and regulation in the future from regulatory agencies in the United States. The technology that appeared to satisfy this requirement was based on water, i.e. aqueous cleaning technology. Several factors played a part in making this choice.

First, the Center had an abundant and inexpensive supply of water. Second, the various aqueous products, i.e. detergents and surfactants, available for use with aqueous processes were either non-toxic or very low in toxicity. Third, aqueous technology materials contained no ODCS . Fourth, aqueous technology materials did not have global warming potential. Fifth, the aqueous products were not classified as "volatile organic compounds" (VOCs) . (VOCs are regulated air contaminants in the United States.) Sixth, the aqueous products could be selected to be biodegradable in the municipal waste water treatment plant servicing the Center. Seventh, the treatment of waste water streams for the removal of various types of contaminants was a proven, mature technology in its own right; this was important because it assured that, if it should become necessary, it would be possible to acquire existing pretreatment technology for the Center's waste water to remove any undesirable contaminants introduced from the Center's cleaning efforts before passing it to the local waste water treatment plant.

When the Center began working with its first aqueous process in 1987, nothing was known at the Center about precision cleaning with water based processes. It soon became evident that not much was known elsewhere, either. Many mistakes were made in those early days of trial and error, but the Center was dedicated to make the concept work. Each mistake or problem was addressed as it was discovered and solutions were found. Over the years that have passed from those early beginnings to the present, the Center has evolved a very sophisticated understanding of aqueous processes and has perfected the technology until it now believes that the vast majority of its cleaning processes, both general and precision, can be converted to aqueous processes. To date, forty-three percent (43%) of the Center's solvent based cleaning processes have been converted to aqueous processes. By January 1, 1995 at least ninety-five percent (95%) will have been converted to aqueous processes.

The Center has installed seventeen aqueous "cleaning centers" throughout its production complex. These seventeen cleaning centers form the basis for the present cleaning process conversions and for all the future conversions to aqueous processes. Each aqueous cleaning center is equipped with a variety of devices. These devices vary from center to center depending upon the processes to be performed. The devices may include an ultrasonic cleaner, a spray booth, a "dish" washing machine, a vacuum oven, Class 100 laminar flow booths, and other miscellaneous items. Incorporated with these well known devices are some extremely important supporting systems. The Center has found these systems to

be critical to the use of aqueous processes for precision cleaning. The systems will be grouped into three categories for explanation, i.e. water supply, water heating, and compressed air drying:

1. Water Supply: The Center has found that the quality of the water used in aqueous cleaning processes for precision cleaning is very important in assuring consistency in spot free cleaning. Each cleaning center is supplied with high quality deionized water meeting the United States' American Society for Testing and Materials (ASTM) Type E2 classification. The water is constantly recirculated through the deionizing system until it is used in the cleaning center. This is done to assure consistency in the quality of the water and to prevent biological film buildup within the system. The deionized water enters the cleaning center with a nominal resistivity of 18 megohms and must meet a 15 megohm threshold for acceptance. As the water enters the cleaning center it is filtered through a 0.2 micrometre absolute filter to remove particulate.

2. Water Heating: The deionized water is heated before use by a point-of-use water heater designed for use with deionized water. With this type of water heater the water is heated as it is used. Since no holding or storage tank is required, there is no loss of the deionization that may occur when deionized hot water sits in a tank. The temperature of the water delivered to the process can be adjusted from 60°F (16°C) to 155°F (68°C) as determined necessary for a particular process.

3. Compressed Air Drying: Drying is an extremely critical operation in precision cleaning if "water spots" and recontamination with small particulate are to be avoided. Drying must occur quickly following the rinsing operation while the parts are still totally damp, and it must be very rapid. The Center accomplishes this in a preliminary drying process using compressed air as a drying medium. For its particular cleaning requirements, the Center has found that the most effective and efficient drying can be accomplished with a hand held blowing device using the compressed air. The blowing device has a specially designed nozzle which permits a technician to use compressed air at a gage pressure of 60 pounds per square inch (414 kilopascals) without any safety concerns relative to the pressure. The pressure and flow rate (approximately 18 cubic feet per minute (0.008 cubic metres per second)) of the air from the hand held device will dry moisture from parts at least as rapidly as CFC-113 will evaporate from the same parts when allowed to air dry in still air. The compressed air has several operations performed on it before it makes contact with the part being dried. It is filtered to remove oil and water until the mass fraction of oil and water is reduced to a level of no more than 0.003 parts per million and to remove particulate larger than 0.01 micrometres. The filtering system used will operate effectively up to gage pressures of 80 pounds per square inch (550 kilopascals) and an air flow rate of 150 cubic feet per minute (0.07 cubic metres per second) . The air is passed through a static dissipating nuclear ionizing device containing the element Po 210. This ionizes the air to nullify any electrostatic charge that may be induced by the technician on the part being dried and, thereby, reduces the incidence of electrostatic attraction of particulate during the drying process. The Center's experience has been that the compressed air works fully as well for drying purposes without heating as it does with heating. As a result, the air is not heated prior to use.

Even with the knowledge and experience gained at the Center from the development and implementation of its aqueous processes, each new application must be carefully examined and tested. It is not unusual that a new application will require tailoring of the various parameters that are part of the aqueous cleaning process. These parameters include, but are not limited to, the type of detergent used, the temperature of the water and drying systems, the water quality, the length of the wash and rinse cycles, the agitation techniques used, the drying techniques used, and the design of the fixture holding the parts being cleaned. One example which illustrates the need for process tailoring concerns the cleaning of an assembly from a device referred to as a gyroscopic compass.

The inner housing assembly from the gyroscopic compass requires precision cleaning prior to repair and, again, prior to final assembly. The assembly consists of the beryllium inner housing of the gyroscopic compass to which a stainless steel motor stator is attached with eight symmetrically placed "tacks" of epoxy. The epoxy is a Bacon Industries product identified as LCA4 and is used with an activator identified as BA5. When this assembly was examined as a candidate for an aqueous cleaning process, it appeared that a process typically used at the Center for beryllium and stainless steel would be acceptable. The process in question required the use of a detergent with the trade name "Versa Clean", ultrasonic wash and rinse cycle times of 3 minutes each, preliminary drying with compressed air and final drying in a vacuum oven for a minimum of one hour. The water temperature during the wash and rinse cycles was 145 $\pm$ 5°F (63 $\pm$ 3°C), and the vacuum oven was operated at a temperature of 150 $\pm$ 5°F (66 $\pm$ 3°C). During the evaluation of the assembly following its first cleaning with the proposed process, it was observed that some of the epoxy tacks had loosened. After a considerable amount of exasperating thought and testing, it was found that the problem was actually quite simple to solve. During the process of installing the motor stator to the inner housing, the epoxy is cured at 97°F (36°C) for 8 hours. Apparently, the glass transition temperature of the epoxy established during the epoxy curing process at 97°F (36°C) was such that the exposure of the assembly to temperatures in the range of 150°F (66°C) during cleaning caused the epoxy to fracture. The temperature of the water in the wash and rinse cycles and the temperature of the vacuum oven used in the final drying process were adjusted to 95 $\pm$ 5°F (35 $\pm$ 3°C). Following this process tailoring, the process worked perfectly.

The Center has had several aqueous processes in full operation for as long as four years. These processes are used as the only means for cleaning on various inertial system parts and assemblies. The various parts and assemblies consist of a variety of materials including jewels, various adhesives and plastics, copper, and alloys of iron, aluminum, and beryllium together with many other materials. Included in these assemblies are over thirty-two different precision instrument bearings (Reference 1). The Center also repairs a variety of electronic circuit cards; many of these are now being cleaned exclusively with an aqueous process to remove the mildly activated rosin (RMA) soldering flux used in the repair process and to generally clean the boards. A specific detergent is used in this process which has undergone extensive testing for its potential short and long term effects on the various metals used in circuit board construction (Reference 2).

The Center <u>never</u> adopts an alternative as a process substitute until it is fully convinced, based upon its testing and evaluation, that the alternative will provide the same quality in the final product as did the ODC based process it replaced. With this understood to be a prerequisite, the Center has made three specific observations from its experiences with the substitution of alternatives:

Observation 1: The quality of the processes has never decreased; in fact, it usually increases. Two examples will be used to illustrate this observation:

Example 1: In the refurbishment processes for the thirty-two different types of precision bearings that the Center restores to original condition for reuse in the systems it repairs, the yield has increased for every bearing (Reference 1). The increase in yield has ranged between twenty-five percent (25%) and sixty-five percent (65%), depending upon the type of bearing. Furthermore, this application of aqueous processes was one of the first at the center and has been in place for over four years. Throughout that time, there has been absolutely no indication in the extensive reliability data the Center maintains on all the systems it repairs, that the long term usefulness of the refurbished bearings has been adversely affected by the use of aqueous processes.

Example 2: The yield from the repair process for a gyroscope consisting predominantly of beryllium increased by 1.5 percent (1.5%) when the ODC based cleaning processes were changed to aqueous processes. The aqueously cleaned gyroscopes have been in use in operating condition for over a year. A number of the gyroscopes, after months of actual operation, have been removed from their parent guidance sets and subjected to an extensive "postmortem" analysis. These preplanned analyses were conducted according to rigorous criteria under the supervision of a team consisting of both government and industry experts. Nothing was found in the postmortem analyses to indicate any adverse, long term effects that could be attributed to the aqueous cleaning processes used in the repair of the gyroscope.

Observation 2: There has been no increased process time following the substitution of an aqueous process for an ODC based process. In fact, the process times are usually reduced. Again, two examples will be used to illustrate this observation.

Example 1: When the cleaning of a gimbal ring used in one of the older aircraft inertial navigation systems repaired at the Center was converted to an aqueous process, the process time for cleaning the gimbal ring decreased by ninety-two percent (92%). The ODC based process for cleaning the gimbal rings was required to be performed manually, one ring at a time. The manual process required about 15 minutes per ring; the aqueous process that was developed permitted 24 rings at a time to be cleaned in 25 minutes. (The cleaning results were also better with the aqueous process.)

Example 2: The total process time for the same gyroscope described in Example 2 of Observation 1 above decreased by 7.1 percent (7.1%) when the cleaning process was converted to an aqueous process. The 7.1 percent (7.1%) reduction in process time for this particular gyro is equivalent to approximately two workdays consisting of eight hours each..

Observation 3: The aqueous processes have been found to be much less expensive than the equivalent processes using ODC solvents. The total cost to convert all of the Center's processes which can be converted to aqueous processes (over ninety-five percent (95%) of the processes) will be approximately US$ 1 400 000. The breakdown of this cost is as follows:

| | | |
|---|---|---|
| -Labor: | None (Used Current Employees) | |
| -Equipment: | | US $ 900 000 |
| -Facilities : | | US$ 400 000 |
| -Deionized Water: | | US $ 100 000 |
| --Total Investment at Completion: | | US$ 1 400 000 |
| -Recurring Annual Cost Estimate: | | US$ 200 000 |

The reduction in use of just the solvent CFC-113 that has resulted from the present conversion of forty-three percent (43%) of the cleaning requirements to aqueous processes is equivalent to an annual reduction in cost to the Center of US$ 1 800 000. This figure is based upon what the Center currently pays for CFC-113. Other cost avoidance the Center has not quantified includes such things as:

- -Cost of methyl chloroform no longer used for cleaning.
- -Discontinued use of motors on CFC-113 spray booth fans.
- -Decreased hazardous waste disposal.

In addition to the three general observations 'discussed above, the Center believes that the total energy consumption for all of its aqueous processes will be no more than and, in fact, probably less than the energy that was used to maintain the ODC solvent based processes. The ODC solvent based system did, indeed, consume large quantities of energy in a variety of ways, Some of the ways are given here. Energy is used to provide the heating and cooling required in the large distillation system used at the Center to reprocess CFC-113 to virgin quality for reuse. Energy is consumed by the large motors on the two carbon vapor adsorption units used to recover ODC vapors from the exhaust air streams of the many ODC spray booths used at the Center.

The carbon vapor adsorption units run constantly throughout the year. The carbon vapor adsorption units use additional energy in the form of steam to purge the carbon beds of adsorbed solvent periodically. Energy is also consumed in the operation of the thirty sump pumps that return used CFC-113 to the distillation system and by the exhaust fans on the CFC-113 impingement spray booths.

Finally, the Center did not ignore the contents of its waste water and the impact those contents may have on the local municipal waste water treatment plant that services the Center. The Center knew the contaminants removed in the general and precision cleaning processes at the Center during the repair of inertial systems were quite small in quantity, and it was felt that both the quantity and the type of contaminants would ultimately not be a problem in the waste water. However, to be certain, it asked representatives of the Environmental Protection Agency of the State of Ohio, USA, to visit on multiple occasions to observe and comment on the waste streams of the processes that have been installed. The results of those visits have been that there are not enough contaminants of any kind being discharged into the waste water treatment plant of the local municipality to warrant any special concern or precaution. In addition, the Center had a study conducted to examine the detergents being used and the contaminants in the waste stream, both at the present time and as projected for the future, to determine if any pretreatment would be necessary in the future (Reference 3). The company that did the study worked closely with the local municipality in conducting their study and the conclusion was that there is no reason for concern if the implementation of aqueous processes at the Center proceeds as is planned. Even with this positive information, the Center remains alert to the condition of its process waste water to forestall any unexpected impact.

## NON-AQUEOUS TECHNOLOGIES

While the Center was committed to making aqueous technology work in its processes, it realized and expected that there would be some cleaning requirements that could not be performed with water and detergent, even with the applied knowledge and experience gained at the Center. This was, in fact, the case. Several specific cleaning requirements have been found which have defied the application of the present capability of aqueous technology; more are expected as the aqueous Process conversion nears completion. These unique requirements will be referred to as non-aqueous cleaning requirements in the remainder of the paper.

It is obvious that non-aqueous cleaning requirements require non-aqueous technologies . As was discussed earlier, the selection of potential alternatives must address the performance requirements of the process, worker safety and health, the environmental impact, and the cost. The

Center has investigated numerous non-aqueous cleaning technologies. From among these, four specific technologies have been identified which the Center feels are essential if it is to meet its non-aqueous cleaning requirements . Each of these technologies appears to meet the Center's performance requirements on the specific cleaning tasks they are being applied to. In addition, the chemicals that form the basis for these technologies have no ozone depleting potential and are either non-toxic or have very low toxicity concerns. These non-aqueous technologies are Alcohol, Methyl Siloxane, Perfluorocarbon, and Supercritical Fluids.

It is expected that these four technologies will provide a solution for the remaining non-aqueous cleaning requirements that may generate. However, each of the non-aqueous solutions has one or more major concerns that will come into play when trying to apply them to remaining non-aqueous cleaning requirements. These concerns include cost (US$), whether or not the material used is regulated as a volatile organic compound (VOC) according to US government requirements, whether or not the material used has direct global warming potential (GWP), whether or not the material used is flammable or combustible, and whether or not the material is classified by the US government as requiring special disposal procedures after use. These concerns for each technology are shown in Table 1 with aqueous technology included for comparison. The concerns given are for the particular materials which will be used at the Center in non-aqueous technologies. These materials will be discussed in more detail in the remainder of the paper. When considering the cost, it is important to acknowledge that at the Center, the material in the aqueous, alcohol, methyl siloxane, and perfluorocarbon technologies, when used for cleaning, may be used for many cleaning operations before it must be replaced. After replacement in the actual process, the used alcohol, methyl siloxane, and perfluorocarbon materials may be economically recovered through distillation. The gases used in the supercritical fluid processes for the Center, i.e. carbon dioxide and ethane, are released directly to the atmosphere after use. In supercritical fluid cleaning processes larger than those intended for the Center, these gases may be economically captured and recycled. In Table 1 the US$/kg figure given for aqueous technology represents the cost for a typical water and detergent solution at the manufacturer's recommended concentration.

## ALCOHOL TECHNOLOGY

One of the non-aqueous cleaning requirements that surfaced at the Center was the cleaning of mildly activated rosin (RMA) flux residue following the installation of a very fragile "pigtail" wire in a gyroscope repaired by the Center for both the US Army and the US Navy. The pigtail wires, in this application, are a form of electrical conductor so designed that when attached between two points that experience minor relative motions, the continuity of the circuit is maintained without contributing any significant restraints to the relative motion. Pigtail connections are made from the gyro case to the float to supply electrical circuit continuity for the pickoff coils, the torquer coils, and the spin motor. The pigtail wire has a maximum diameter of 0.001 inch (25.4 micrometres) and is 99.4 percent (99.4%) silver with a small amount of nickel and magnesium. The wire is coiled to form a small diameter (approximately 0.03 inch (762 micrometres)) spring that has the lowest spring rate possible.

This pigtail wire is so fragile that the surface tension of water distorts and deforms it. This simple fact makes it impossible to submit the assembly containing the pigtail to any form of aqueous cleaning. The existing process uses MCF to remove the flux and CFC-113 to rinse the assembly. The assembly is immersed in both the MCF and the CFC-113 in this process, but neither solvent causes any deformation of the pigtail.

The alternative cleaning process that has been found is a process using isopropyl alcohol. The isopropyl alcohol has a surface tension of 21.3 dynes per centimetre (21.3 millinewtons per metre) which is between the surface tension of CFC-113 (17.3 dynes per centimetre (17.3 millinewtons per metre)) and MCF (25.5 dynes per centimetre (25.5 millinewtons Per metre)) . Testing has shown that immersion in isopropyl alcohol during the cleaning process does not deform the pigtail. It also is a fact that isopropyl alcohol is an acceptable solvent for RMA flux. Another favorable aspect of using isopropyl alcohol for precision cleaning is that there is a considerable body of data from Europe on its use as a precision cleaning fluid including validation of compatibility with a variety of metals, plastics, and adhesives (References 4 and 5) .

The primary concern (see Table 1) with the use of isopropyl alcohol is its flammability. Its flash point is 53°F (11.7°C). Other concerns are that it is considered a VOC and is subject to regulation and that it has to be disposed of with special procedures since it is a flammable liquid.

The Center is in the process of buying a cleaning system similar to the one in which the testing mentioned above was done (Reference 6) . It is expected to be installed in January, 1994. This system has proven design features which meet the stringent requirements of the fire, safety, and environmental concerns of the US Air Force as interpreted by the Center's enforcement officials. The system is designed to clean with pure isopropyl alcohol with or without ultrasonics and, even though it contains a flammable liquid, the safety features and design of the system are such that it may be placed in a normal production area without requiring any additional precautions in electrical wiring or construction. Among the features of the system are a "concentrator" for impurities in the alcohol bath and a "scrubber" for the exhaust air from the system. The concentrator constantly removes impurities from the alcohol bath and returns pure alcohol in the form of alcohol vapor to the vapor zone above the bath where it is condensed back into the bath. The water-based scrubber removes the alcohol vapors and, hence, the VOC emissions from the exhaust air stream from the process to the low level of less than 0.04 pound per hour (5 milligrams per second) . The water from the scrubber with typical operation of the cleaning system will contain a mass fraction of alcohol less than 200 parts per million. Because of this low concentration, the current position of the Center is that the water from the scrubber may be discharged directly into the Center's waste water without pretreatment. Should, in the future, pretreatment be required, the waste water stream from the scrubber could be easily captured and appropriately processed.

Another difficult cleaning requirement at the Center is the removal of very viscous suspension and damping fluids from parts with complex geometry. Some of these fluids, such as polybromotrifluoroethylene, are very dense and difficult to remove with the Center's aqueous processes. Preliminary testing has shown that polybromotrifluoroethylene can easily be removed from some of the Center's more complex parts by the isopropyl alcohol cleaning process. If further testing is satisfactory, the alcohol system may be adapted to this cleaning operation. Some of the assemblies containing the dense fluids are very fragile in their construction. This fragility causes them to be very susceptible to damage from even minor agitation. When this combination of non-aqueous cleaning requirements is taken together, the advantages of alcohol cleaning become apparent.

## METHYL SILOXANE TECHNOLOGY

One of the non-aqueous cleaning requirements at the Center is the removal of silicone based damping fluid, specifically phenylmethyl silicone, from the gyro parts in which it is used.

The Center is investigating the use of volatile methyl siloxane fluids as a medium for the removal of this material. These fluids have some attractive features (Reference 7) including their distinctive ability to remove phenylmethyl silicone. They also have some concerns in addition to the ones listed in Table 1. preliminary evaluations have shown these materials to be compatible with the specific metals and other materials of the parts to be cleaned, but there are insufficient test results to make a conclusive decision on this issue. Also, there is a question of the stability of the fluids in actual use. These two concerns are being addressed through further testing at the Center.

One of these materials, 0S-10 made by Dow Corning Corporation, has been used quite successfully for certain cleaning operations in the same system being purchased by the Center for isopropyl alcohol cleaning. (0S-10 has a flash point of 30°F (-1°C) so it is quite flammable.) The new cleaning system will serve as a test-bed for the evaluation of some of the methyl siloxane fluids for other non-aqueous cleaning requirements at the Center. The fact that the methyl siloxanes may be de-listed as VOCS by the US Environmental Protection Agency in the near future is a positive factor for the fluids. Because of the VOC issue, it may be desirable to use them in place of alcohol if they demonstrate the same cleaning ability as alcohol and the same degree of compatibility with the materials being cleaned. On the other hand, the methyl siloxanes are currently much more expensive than isopropyl alcohol. These kinds of issues must be dealt with as the Center's experience with the two technologies evolves.

## SUPER CRITICAL FLUID TECHNOLOGY

The very critical precision cleaning required in the repair of inertial navigation and guidance systems and a strong concern about the impending phaseout of ODCs, the primary cleaning solvents traditionally used for this purpose, stimulated the use of the small Business Innovative Research program to assist in developing solutions. The Small Business Innovative Research program funding is made available to the US Department of Defense (DoD) by the US Congress as a mechanism to encourage small US businesses with knowledge and innovative technology to apply their capabilities to solve DoD problems and, in so doing, become a viable part of the US technology base. Two such Small Business Innovative Research projects were initiated in 1991 with the Aerospace Guidance and Metrology Center being the recipient of the deliverable technologies.

One of the projects dealt with perfluorocarbon technology and will be discussed in the next section of this paper. The other project dealt with the application of supercritical fluids technology to solve difficult cleaning problems in inertial components.

Supercritical fluids were not new, but the application of this technology to precision cleaning of inertial components was an unexplored concept when the Small Business Innovative Research project was begun (Reference 8). The company selected to do the project was the Phasex Corporation of Lawrence, Massachusetts, USA. The Phasex Corporation, while considered a small business, had considerable experience in the application of supercritical fluid concepts. The cleaning of complex parts using supercritical fluids is a direct outgrowth of their prior experience in fundamental and practical volubility phenomena and materials interactions.

TABLE 1

| Technology | US$/kg | VOC? | Direct GWP? | Flammable/ Combustible | Special Disposal? |
|---|---|---|---|---|---|
| Aqueous | 0.22 | No[1] | No | No | No[4] |
| Alcohol | 3.31 | Yes | No | Yes | Yes |
| Methyl Siloxanes | 17.64 | Yes[2] | No | Yes | Yes |
| Perfluorocarbons | 37.48 | No[1] | Yes | No | No |
| Supercritical Fluids | | | | | |
| Carbon Dioxide | 0.13 (2.00[5] | No[1] | Yes[3] | No | No |
| Ethane | 3.97 (20.00)[5] | No[1] | No | Yes | No |

1 By US Environmental Protection Agency definition.

2 An exemption as a VOC is under consideration by the United States Environmental Agency and is expected to be granted in the near future.

3 Carbon dioxide does have direct global warming potential, but the carbon dioxide gas available from the commercial market for industrial use is extracted from the waste streams of chemical manufacturing processes and coal burning power plants . As such, it is currently viewed in the United States as an acceptable emission from a "secondary" user's perspective.

4 While aqueous products, themselves, may require no special disposal procedures, the aqueous waste stream might. This would be dependent upon the nature and volume of the contaminants removed during the aqueous cleaning processes.

5 Total cost of the gas used in a typical application for one cleaning operation in the supercritical cleaning machine at the Center. This cost assumes the cleaning chamber is full of parts and all the gas is vented from the machine to the atmosphere. With larger supercritical fluid systems, it may be economical to recover and recycle the gas which could reduce this relative cost.

TABLE 1

One of the specific non-aqueous requirements that the Center worked with Phasex to resolve was the removal of phenylmethyl silicone oil from a particularly complicated assembly. The assembly in question was part of an accelerometer and consisted of a small housing made of aluminum containing a pendulum made of beryllium copper alloy, an iron core magnet, and pigtails made from predominantly silver ribbon. The pigtails are approximately one inch (2.5 centimetres) long and are 0.0003 inches (7.62 micrometres) thick by 0.004 inches (102 micrometres) wide. Portions of the assembly are attached using LCA4 epoxy with a BA5 activator. (Both LCA4 and BA5 are Bacon Industries products.) The phenylmethyl silicone oil is forced around these various assembly components during operation, and its removal during cleaning is complicated by the fact that the spacings between the various components are very small. There are also recessed spaces that are difficult to access. It is preferred to clean the assembly fully assembled. This is difficult to do even with the CFC-113 and impossible to do with an aqueous process.

The past experience of Phasex was a considerable asset in shortening the time it took the company to solve the problem. The volubility in supercritical fluids (SCFs) of silicone oils and siloxane polymers of various molecular weights was investigated by Phasex in the mid 1980s. During these studies it was shown that carbon dioxide was effective for dissolving these materials only up to limited molecular weights depending upon their specific chemical compositions and/or functionalities. Beyond these molecular weight levels, it was found that ethane more effectively dissolved the silicone oils and siloxanes up to molecular weights as high as 500 000.

When challenged with cleaning the accelerometer housing, Phasex considered both carbon dioxide and ethane as potential candidates for removing silicone oils with the SCF process. The molecular weight of the oil was unknown but the Center furnished the company a sample for solubility testing. Carbon dioxide was tested at temperatures of 140°F (60°C) and 176°F (80°C) and at various pressures ranging up to a gage pressure of 7500 pounds per square inch (51.7 megapascals). With carbon dioxide at a gage pressure of 7500 pounds per square inch (51.7 megapascals), only about sixty percent (60%) of the phenylmethyl silicone oil could be dissolved, even at a solvent-to-feed ratio of 2000:1. Based upon previous tests with dimethyl, phenylmethyl, and diphenyl silicone fluids, ethane was tested as a solvent. At a gage pressure of 6000 pounds per square inch (41.4 megapascals) and 176°F (80°C), a solvent-to-feed ratio of only 400:1 dissolved one hundred percent (100%) of the phenylmethyl silicone oil. Thus, ethane was the preferred supercritical solvent for removing this rather high molecular weight silicone oil from the accelerometer housing.

Extensive testing was done by Phasex (Reference 9) to validate the compatibility of carbon dioxide and ethane with the various materials of the special assemblies from which the Center had difficulty removing certain oils. The combination of Phasex's experience, the testing and investigation they did on the Small Business Innovative Research project, and their close collaboration with the Center's process experts has been incorporated into the design and construction of an SCF cleaning station. This cleaning station was installed at the Center in November, 1993 and operates on either carbon dioxide or ethane. It has a chamber 4 inches (10.16 centimetres) in diameter and 12 inches (30.48 centimetres) deep in which the parts to be cleaned are placed. The cleaning station is safe and easy to use and delivers a superb product.

It must be remembered that SCF technology has not yet been demonstrated to be a truly effective means of removing particulate. It is, however, excellent at removing oils and greases from certain parts and assemblies where the temperature and pressure of the process will cause no harm.

The SCF cleaning station is intended to solve specific problems. It is not intended to be used where other technologies can do an equal or better job faster and cheaper and with an acceptable environmental impact .

## PERFLUOROCARBON TECHNOLOGY

The second Small Business Innovative Research project was initiated to develop a particulate removal capability equivalent to that of CFC-113 using perfluorocarbon technology. The company selected to do this was Entropic Systems Inc. of Winchester, Massachusetts, USA. Entropic Systems Inc., as was the case with the Phasex Corporation, had had considerable experience in their field of expertise, i.e. particulate removal with emphasis on the use of perfluorocarbons. In the Small Business Innovative Research project, Entropic Systems' challenge was to apply that knowledge and experience to certain critical requirements encountered by the Center in its repair of inertial systems and components. Considerable effort was expended by Entropic Systems in collaboration with Center personnel in testing and evaluating various processes and process parameters (Reference 9) .

The results were extraordinary. Entropic Systems developed an ultrasonic process to remove particulate using a perfluorocarbon, such as perfluoroheptane (PF-5070 made by 3M Corp.), together with a fluorinated surfactant, such as purified Krytox 157FS (made by DuPont Corp.). This combination was very carefully tested and conclusively shown to remove particulate as well as CFC-113; actually, it removed particulate much better than CFC-113.

The process has been incorporated into a cleaning station which will be delivered and put into operation at the Center in early 1994 (Reference 10). The cleaning station is designed to have extremely low emissions, on-line particle sensors and an ultra violet spectrophotometer to monitor cleaning in real time, and many other features to make it a valuable tool for very critical particulate removal requirements.

While the perfluorocarbons used in the process will easily remove fluorinated oils, they have little solvency for other organic contaminants. Consequently, for some applications, the cleaning station may be used as a final cleaning operation for the specific purpose of removing particulate where high quality particulate removal is necessary.

The Center is very much aware of the extremely high global warming potential of the perfluorocarbons and of the US Environmental Protection Agency's concern for their use. The Center shares that concern and will use the perfluorocarbon technology only where nothing else will achieve the desired particulate removal necessary to the functioning of certain very high precision inertial components. However, with prudent care and until something better is found, this technology offers an important contribution to the effort to totally eliminate dependence on ODCs where high precision particulate removal is required.

## CONCLUSIONS

The challenge of substituting alternatives for the precision cleaning processes using ODCs for general and precision cleaning is not an easy one. The difficulty often lies in selecting the "best" technology from among the many available technologies that form the alternatives. The best technology depends upon many factors related to a particular industry's needs. Also, more than one "best" technology is often required to provide a complete solution to an industry's ODC elimination problems.

An important fact to keep in mind in this effort is that there have been many success stories around the world for ODC elimination. What is being done at the Aerospace Guidance and Metrology Center is just one of those success stories. For those who have not yet implemented alternatives for ODC processes, these success stories offer, through the sharing of information, the ability to move past many of the difficulties that those with the success stories had to overcome.

This has the potential to greatly expedite the selection of a "best" alternative and to do it at a reduced overall cost.

The discussion in this paper, of necessity, is extremely brief and does not address the considerable detail that has gone into the development, testing, and application of each of the technologies for the cleaning needs of the Aerospace Guidance and Metrology Center. However, anything that the Center has learned or done in this area in the past and what it will learn and do in the future will be freely shared with any interested organization. Upon request, the author will discuss how to obtain this information and how to arrange for a site visit to the Center, if one is desired.

The US Air Force, through its role as an affiliate member of the international organization known as the Industry Cooperative for Ozone Layer Protection (ICOLP), has committed to share the knowledge and experience it has gained in eliminating ODCs from its many diverse processes. The Aerospace Guidance and Metrology Center is proud to play a role in that commitment.

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